

PCTWORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification⁶ : C22F 1/04, 1/057	A1	(11) International Publication Number: WO 96/18752 (43) International Publication Date: 20 June 1996 (20.06.96)
(21) International Application Number: PCT/GB95/02878 (22) International Filing Date: 11 December 1995 (11.12.95) (30) Priority Data: 9424970.3 10 December 1994 (10.12.94) GB (71) Applicant (for all designated States except US): BRITISH AEROSPACE PUBLIC LIMITED COMPANY [GB/GB]; Warwick House, Farnborough Aerospace Centre, P.O. Box 87, Farnborough, Hampshire GU14 6YU (GB). (72) Inventor; and (75) Inventor/Applicant (for US only): PRICE, Howard, James [GB/GB]; British Aerospace Regional Aircraft Limited, AVRO International Aerospace Division, Woodford Aerodrome, Chester Road, Woodford, Cheshire SK7 1QR (GB). (74) Agent: EDIS, Ronald, Malcolm; British Aerospace public limited company, Corporate IPR Dept., Lancaster House, P.O. Box 87, Farnborough Aerospace Center, Farnborough, Hampshire GU14 6YU (GB).		(81) Designated States: AU, BR, CA, CN, JP, KR, MX, PL, RU, SG, UA, US, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report.</i>
(54) Title: HEAT TREATMENT OF ALUMINIUM-LITHIUM ALLOYS (57) Abstract A method of heat treating an aluminium-lithium alloy is provided. The method includes carrying out a succession of at least two artificial ageing steps. The first such step is carried out within a first temperature range and one or more further steps are carried out within successively reduced temperature ranges to promote the precipitation of the δ' phase of the alloy.		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	GB	United Kingdom	MR	Mauritania
AU	Australia	GE	Georgia	MW	Malawi
BB	Barbados	GN	Guinea	NE	Niger
BE	Belgium	GR	Greece	NL	Netherlands
BF	Burkina Faso	HU	Hungary	NO	Norway
BG	Bulgaria	IE	Ireland	NZ	New Zealand
BJ	Benin	IT	Italy	PL	Poland
BR	Brazil	JP	Japan	PT	Portugal
BY	Belarus	KE	Kenya	RO	Romania
CA	Canada	KG	Kyrgyzstan	RU	Russian Federation
CF	Central African Republic	KP	Democratic People's Republic of Korea	SD	Sudan
CG	Congo	KR	Republic of Korea	SE	Sweden
CH	Switzerland	KZ	Kazakhstan	SI	Slovenia
CI	Côte d'Ivoire	LI	Liechtenstein	SK	Slovakia
CM	Cameroon	LK	Sri Lanka	SN	Senegal
CN	China	LU	Luxembourg	TD	Chad
CS	Czechoslovakia	LV	Latvia	TG	Togo
CZ	Czech Republic	MC	Monaco	TJ	Tajikistan
DE	Germany	MD	Republic of Moldova	TT	Trinidad and Tobago
DK	Denmark	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	US	United States of America
FI	Finland	MN	Mongolia	UZ	Uzbekistan
FR	France			VN	Viet Nam
GA	Gabon				

HEAT TREATMENT OF ALUMINIUM-LITHIUM ALLOYS

This invention relates to the heat treatment of aluminium-lithium alloys and in particular to such heat treatment for the strengthening of such alloys and for the optimisation of such alloys' plane stress fracture toughness. Such alloys are known in particular for use in aircraft skin construction, and more particularly for commercial aircraft fuselage, wing and empennage construction. In this application in particular the low density, high stiffness and excellent fatigue properties of aluminium-lithium alloys enable weight savings to be achieved to maximise profitability of the aircraft.

Prior Art references which are relevant to this invention known at the time of the invention are as follows. "Effect of thermal exposure at 70°C on the performance of damage tolerant aluminium-lithium alloy sheet." February 1995. Reference DRA/SMC/WP952008 by D.S. McDarmaid; "Mechanical properties of 2024-T3 aluminium alloy sheet. December 1991. Reference TR91071 by D.S. McDarmaid, C.E. Thomas and C. Wheeler.

The aluminium-lithium (Al-Li) alloys registered with the ALUMINUM ASSOCIATION as AA8090 and AA2091 (hereinafter referred to without the "AA" prefix) in recrystallised sheet form and under-aged tempers have been shown to possess attributes of "Damage Tolerance" in that

fatigue crack growth rates are commendably slow coupled with reasonably high levels of plane stress fracture toughness (K_{IC}). As such, both products have been extensively investigated as potential alternatives to the currently most widely used materials for civil aircraft skin applications, in particular for fuselages eg alclad 2024 T3 and 2014A T4 sheet, where the density reduction associated with the lithium-bearing alloys would enable considerable amounts of weight to be saved. 8090 in plate form has also been investigated for upper and lower wing skin and empennage applications and may also be considered for upper wing skins.

In addition to the requirement for damage tolerance there are several other necessary characteristics which any new skin material and particularly fuselage wing and empennage skin materials must possess. These include adequate strength, good corrosion resistance and an often unstated but very important requirement of long-term thermal stability ie the ability to withstand prolonged periods at moderately elevated temperatures without an appreciable or unacceptable loss in any of the key attributes. For a sub-sonic civil aircraft fuselage the worst case from a consideration of thermal instability involves on the ground exposures to the combined effects of high ambient temperatures and intense solar radiation. It is generally

accepted that in tropical conditions fuselage skin temperatures of up to 70-85°C can be achieved when the sun is at or near its zenith. Over the life of an aircraft this could, in the worst case, represent a cumulative high temperature exposure of approximately 65000 hours (eg 6 hours per day for 30 years) although such an exposure would only be achieved for aircraft either stored in desert conditions or operated irregularly from tropical bases. Thermal stability is also one aspect of concern when considering the use of Al-Li alloys for wing and empennage skin applications.

The 8090 and 2091 alloys have been primarily investigated for fuselage skin applications in the T81 and T84 conditions respectively. The T81 condition for 8090 is achieved by artificial age hardening ("ageing") from the T31 condition (ie solution treated and controlled stretched) for 24 hours at 150°C whilst the T84 condition for 2091 is achieved by ageing from the T3 condition for 12 hours at 135°C following a slow ramp up from ambient to 135°C. These treatments are intended to produce products which mimic the mechanical properties of alclad 2024 T3 (ie the lower limit for 0.2% Proof Stress has been set as approximately 270 MPa) in order that substitutional applications can more easily be considered. There is, also, the widespread perception that Al-Li alloys require static strengths at least equivalent to alclad 2024 T3 to

be successful in the fuselage skin application. This is not necessarily so since the increase in Young's Modulus associated with the lithium content is capable of more than off-setting any slight reduction in strength which might now be seen to be required in order to properly satisfy a real requirement for very high fracture toughness and good impact resistance.

Despite the use of artificial ageing treatments, both the Al-Li products referred to are known to lack thermal stability in the temperature range 70-85°C and an increase in strength coupled with a disproportionately large reduction in Kc results after relatively short isothermal exposures (ie a very significant effect after 1000 hours). This inverse relationship between strength and Kc for Al-Li alloys has been demonstrated on many occasions. Given that the initial toughness levels for both alloys aged to their respective prior art conditions (ie T81 and T84 for 8090 and 2091 respectively) are marginal for the intended application when compared to alclad 2024 T3 (the current industry standard) this absence of thermal stability and the pernicious effect on toughness of even apparently very small increases in strength is widely regarded as a major contributory factor accounting for the current lack of any significant civil aircraft fuselage applications.

The cause of thermal instability is attributed to an on-going precipitation of δ' (Al,Li). The reason for the continued precipitation of δ' , and hence the thermal instability, is that there is an inverse relationship between the equilibrium volume fraction of δ' and temperature (ie the equilibrium volume fraction increases as temperature is reduced). The high rate of diffusion of lithium in aluminium ensures that the formation of δ' is not effectively diffusion rate controlled until the temperature falls some considerable way below the exposure temperature of concern. It therefore follows that even extensive ageing at the stated prior art ageing temperatures (ie 135-150°C) will never achieve anything approaching a complete precipitation of δ' and a high thermodynamic driving force for on-going precipitation, coupled with adequate rates of lithium diffusion, will exist at temperatures at or close to (below) the maximum thermal exposure temperatures considered. Instead, extensive ageing at these "higher" temperatures only serves to increase the volume fraction of other phases such as S' (Al,CuMg) leaving a structure overly high in strength but relatively low in δ' . Subsequent long-term thermal exposure therefore results in a large increase in

the δ ' volume fraction, an increase in strength and embrittlement.

To illustrate the effect of on-going δ ' precipitation duplicate samples of a batch of (hereinafter referred to as "Batch 1" material) 8090 T81 were given a range of thermal treatments prior to being exposed to an elevated temperature for a considerable length of time. The composition in weight percent of the Batch 1 material was:

Li	Cu	Mg	Fe	Zr	Al
2.23	1.14	0.79	0.045	0.06	Remainder

The treatments chosen included a 10 minute "reversion" at 200°C from the T81 condition (ie causing a drop in 0.2% Proof Stress due to δ ' dissolution), followed by a re-age of 170°C for 4 hours (ie to achieve a recovery to approximately the original level of T81 0.2% Proof Stress and, finally, an extensive over-ageing treatment of 220°C for 12 hours in addition to the T81 initial treatment.

After tensile testing one long transverse (LT) oriented sample representative of each condition the duplicate samples of all conditions including the T81 "Control" condition were then exposed for 920 hours at 100°C in order to crudely represent a lifetime's exposure to tropical temperatures. The results of the mechanical

property tests and electrical conductivity measurements made are shown in Table 1.

It is clear from Table 1 that the on-going precipitation at 100°C results in a considerable increase in strength. The reverted material recovers to a higher strength than is the case for the Control condition indicating the ineffectiveness of reversion as a means of increasing the toughness of 8090 where consideration must also be made of thermal instability effects since the initial benefit of reversion is short-lived and the treatment can, ultimately, be expected to be harmful as it results in a higher final strength after thermal exposure. The increase in strength of the reverted material over and above the un-reverted material at the conclusion of the thermal exposure is attributed to the additional S' precipitated during the reversion process. Similarly, the additional increase in strength of the reverted and re-aged material following thermal exposure compared with either of the T81 and T84 plus reversion conditions is attributed to the increased S' associated with 4 hours at 170°C.

Finally, the use of over-ageing is seen to be completely ineffective at achieving stability with a 48 MPa rise in 0.2% Proof Stress being apparent at the conclusion of the 920 hour exposure. Similar results for all starting conditions would be anticipated for exposure

at, say, 70°C and an even higher equilibrium volume fraction of δ' would be realisable at this temperature than at 100°C although the exposure time required to achieve saturation would be that much greater at the lower temperature due to the reduced diffusion rates.

It should be noted that the Batch 1 8090 sheet had a T81 LT 0.2% Proof Stress of 293 MPa and which then reached what is believed to be a δ' -saturated 0.2% Proof Stress of 320 MPa following 920 hours thermal exposure at 100°C, ie a rise of 27 MPa.

According to the invention an improved method of heat treating Aluminium-Lithium alloy includes carrying out a succession of at least two artificial ageing steps, the first such step being carried out within a first temperature range and at least one further step being carried out within a successively reduced temperature range.

The specific promotion of δ' precipitation is thus achieved and for appropriately selected temperature ranges capping of the S' volume fraction is achieved in conjunction with this to attain a condition of use with adequate but not excessive initial strength which is compatible with the requirement of high fracture toughness, with the ability to retain adequate fracture toughness following long term exposure to moderately elevated temperatures. Where other appropriate temperature

ranges are selected according to the invention it is possible to combine the promotion of δ' precipitation with high levels of S' volume fraction thereby resulting in a level of strength that is higher than otherwise would be possible for an alloy of this composition for a given total ageing treatment time.

The conclusion reached was that thermal stability at, say, 70-85°C can only be achieved by the realisation of an equilibrium volume fraction of δ' for this temperature. The achievement of δ' saturation needs to be achieved without realising too high a 0.2% Proof Stress level which would otherwise be incompatible with the omnipresent requirement for high fracture toughness.

Ageing trials according to the invention were then conducted using an 8090 T31 starting condition material which was arrived at by re-solution treatment and controlled stretching of some Batch 1 8090 T81 material. NB Re-solution treatment was carried out at 505°C to avoid grain growth. Ageing commenced at 150°C but for a short duration (very much less than the prior art 24 hours at 150°C) followed by progressive reductions in temperature and increases in ageing time in order that the volume fraction of S' and phases other than δ' could be capped and a high volume fraction of δ' realised.

In this way it is now believed that a condition with a superior balance between δ' and S' precipitate volume fractions and precipitate size distribution can be achieved with a relatively low level of 0.2% Proof Stress (and, hence, high fracture toughness) and with limited capacity for further strengthening by on-going precipitation of δ' .

The adoption of this form of retrogressive step-wise (RS-W) ageing treatment according to the invention fully recognises the need to precipitate sufficient S' to prevent what would otherwise be a plastic deformation mechanism dominated by intense planar slip - a deformation mechanism which would, if not properly inhibited by the presence of S' , then result in low levels of ductility, particularly in the longitudinal direction.

During this initial work with re-solution treated Batch 1 material a large number of temperature/time RS-W ageing combinations were studied. Of particular note were treatments based around a 4-step RS-W ageing sequence commencing with either 1 hour or 3 hours at 150°C followed by periods at 135°C, 120°C and 100°C as shown below:

1 hour/150 + 6/135 + 3/120 + 50/100°C (see Table 2A)

1 hour/150 + 6/135 + 8/120 + 50/100°C (see Table 2B)

1 hour/150 + 6/135 + 16/120 + 50/100°C (see Table 2C)

1 hour/150 + 12/135 + 6/120 + 50/100°C (see Table 2D)

1 hour/150 + 12/135 + 16/120 + 50/100°C (see Table 2E)

3 hours/150 + 12/135 + 6/120 + 50/100°C (see Table 2F)

3 hours/150 + 6/135 + 16/120 + 50/100°C (see Table 2G)

These treatments and the resulting mechanical properties and electrical conductivity results, both during the ageing sequence and as a result of various periods of thermal exposure at 85°C and 70°C, are shown in Tables 2A-2G.

Subsequently, a new batch of 8090 sheet was obtained (hereinafter referred to as "Batch 2") which had not been previously solution heat treated. This material was used for solution heat treatment and ageing trials in order to optimise the process of RS-W ageing. The composition in weight percent of the Batch 2 sheet material was:

Li	Cu	Mg	Fe	Zr	Al
2.26	1.21	0.69	0.047	0.06	Remainder

From the results of the Batch 1 trials it was realised that the 135°C step was apparently resulting in excessive ageing of the non- δ' phases and so might be discontinued. It was also recognised that if the fuselage

structure was to be adhesively bonded (ie the attachment of stringers to skins) then either a 150°C or a 120°C curing resin system such as REDUX (registered trade mark) 775 (CIBA) or AF163-2 (3M), or similar, would most likely be used. In the case of REDUX 775 (150°C cure) the cure cycle could be combined with the 150°C RS-W ageing step and all subsequent steps would then be applied to the bonded skin/stringer assembly. In which case there would be an economic advantage in reducing the temperature of the second step such that the assembly would not require an over-pressure to protect the (phenolic) adhesive. This would be achieved by reducing the temperature of the second step from 135°C to 125-120°C whereas the continued use of a 135°C ageing step would necessitate that this ageing step took place in an autoclave or bonding press. If a 120°C cure resin system such as AF163-2 was to be used then the cure cycle could be introduced after completion of all ageing steps of greater than 120°C. No over-pressure would be required for any selection of ageing temperature equal to or less than 120°C.

A series of RS-W ageing trials was undertaken using Batch 2 material which had been solution treated at 530°C and controlled stretched $1.75\% \pm 0.25\%$. Of note are the following RS-W treatments:

1 hour/150 + 6/135 + 8/120 + 50/120°C (included to bench-mark Batch 2 material with Batch 1) (See Table 3A)

1 hour/150 + 8/120 + 24/105 + 24/95°C (See Table 3B)

1 hour/150 + 16/120 + 24/105 + 24/95°C (See Table 3C)

1 hour/150 + 8/125 + 24/105 + 24/95°C (See Table 3D)

1 hour/150 + 16/125 + 24/105 + 24/95°C (See Table 3E)

1 hour/135 + 8/120 + 24/105 + 24/95°C (See Table 3F)

1 hour/135 + 16/120 + 24/105 + 24/95°C (See Table 3G)

2 hour/120 + 32/105 + 24/95°C (see Table 3H)

8 hour/120 + 24/105 + 24/95°C (see Table 3J)

These trials showed that the 135°C step was superfluous and that a direct transition from about 150°C to about 120°C (or 125°C) was preferable. The treatments commencing at 135°C and 120°C had some merit but produced a fully heat treated condition that was low in strength but which ultimately, on thermal exposure, rose to levels comparable with the treatments which commenced at 150°C and so there was expected to be no benefit in terms of usable toughness.

On the basis of the tensile test data from the above tests the sequence 1hour/150°C + 8/120°C + 24/105°C + 24/95°C was selected for further investigation and refinement. This included ageing full-sized sheets to enable wide panel fracture toughness testing to be carried out.

The result of the first fracture toughness test carried out on 1.9mm thick Batch 2 material aged 1 hour/150°C + 8.120°C + 24/105°C + 24/95°C is shown in Figure 1 in the form of a fracture resistance curve (R-curve). The result is compared to R-curves applicable to prior art 8090 T81 and reverted 8090 T81 (Reference 1), an unstable condition previously shown to produce improvements in toughness together with alclad 2024 T3 (Reference 2).

It can be seen that the application of the RS-W treatment of the invention has produced a condition of very high toughness and which is comparable to, or better than, alclad 2024 T3. This is the first known reported occurrence of 8090 sheet exceeding the toughness of alclad 2024 T3. A second 1.9mm thick 8090 sheet was given the above RS-W treatment followed by 2000 hours thermal exposure at between 70°C and 75°C. The R-curve for this material is shown in Figure 2 together with the un-exposed R-curve. Also shown is an R-curve for prior art 8090 T81 material with and without 2000 hours thermal exposure at 70°C (Reference 1). It can be seen that although the RS-W

SUBSTITUTE SHEET (RULE 26)

material has suffered a reduction in toughness the reduction (approximately 6%) is very much less and from a much higher starting level than was the case for prior art 8090 T81.

NB: The comparative data extracted in graphical form from References 1 and 2 is presented for illustrative purposes only and is not intended to limit the invention.

Trials were also conducted to determine sensitivity to temperature and time variations for the first ageing step and to determine whether the final step of 24 hours/95°C could usefully be truncated. The results of these trials are shown in Tables 4A, 4B and 4C for Batch 2 material.

It was established that the first step could be shortened to 0.75 hours or extended to 1.25 hours without undue deleterious effects being apparent. It was also found that the final step could be truncated to 8 hours for material given 1 hour/150°C or 1.25/150°C without a significant effect on the final strength being apparent and, for applications where strength is not critical, this step can be omitted completely and/or the shorter 150°C ageing treatment adopted. The preferred ageing treatment identified as a result of this work is:

1 hour/150°C + 8/120°C + 24/105°C + 8/95°C

The 4-step treatment has the advantage of maximising the degree of benign strengthening (ie strengthening due to δ' precipitation) without requiring an overly long ageing treatment which might be uneconomic.

The treatment was found to be reasonably insensitive to ageing temperature within the range $\pm 5^\circ\text{C}$ (all steps) and to variations in the length of individual treatments within the range $\pm 25\%$ of the stated time.

This preferred ageing treatment was also found to engender optimum resistance to intergranular corrosion as measured by the ASTM G110 corrosion test with depth of corrosion penetration limited to approximately $150\mu\text{m}$ and with a tendency to form localised corrosion pits with very little or virtually no intergranular attack present. This is in very marked contrast to 8090 T81 which often exhibits in excess of $250\text{--}300\mu\text{m}$ of attack and which is characterised by an extended network of intergranular penetration. The forms of intergranular attack for the RS-W and T81 conditions are shown in Figures 3 and 4 respectively.

Several more full-sized sheets were then given the preferred ageing treatment of 1hour/150 + 8/120 + 24/105 + 8/95°C. These sheets were intended to establish the initial toughness level for 1.6mm sheet and to provide specimens for long-term thermal exposure such that the

R-curves of thermally sensitised material could be determined. The results of an R-curve test on this material in the fully heat treated condition are shown in Figure 5. The R-curve is slightly lower than for the 1.9mm material and the difference is considered to be due to the rolling schedule associated with 1.6mm gauge, to differences in lithium depletion, to a thickness effect per se, or to a combination of these effects.

A sheet of Batch 2 material sufficient for a large number of tensile tests has been given the preferred ageing treatment and has completed a 2000 hour thermal exposure test at 70°C together with comparative Batch 2 material initially aged to the T81 condition. The results are shown in Table 5 and plotted as 0.2% Proof Stress versus Log_{10} Exposure Time in Figure 6.

It is apparent from Figure 6 that the T81 material has undergone an incubation period from approximately the 100 hours exposure point to somewhere slightly in excess of the 1000 hour exposure point during which virtually no change in 0.2% Proof Stress was apparent. There was then a rapid increase in 0.2% Proof Stress. In contrast, the RS-W aged material exhibited no such incubation effect and a steady rise in 0.2% Proof Stress versus Log Exposure Time is evidenced. It should be noted that the gradient of the two curves (excluding the incubation period for T81) appears almost identical thereby indicating that the

"advantage" of lower strength in the RS-W material is being maintained and extrapolation to the 65000 hours point suggests that the T81 material would ultimately age to a 0.2% Proof Stress of approximately 349 MPa whereas that of the RS-W material would not exceed approximately 318 MPa. This represents an improvement in terms of preventing a strength increase of approximately 31 MPa that would otherwise occur.

However, this final predicted 0.2% Proof Stress level for Batch 2 RS-W material is regarded as approximately 25-30 MPa above a value considered compatible with a target of matching the plane stress fracture toughness of alclad 2024 T3. To achieve a further reduction in the level of the δ' -saturated 0.2% Proof Stress may require a compositional adjustment to be made in combination with the RS-W treatment. For the 8090 alloy it is believed that the magnesium level should be reduced from the 0.69% level present in Batch 2 to substantially the minimum level in the compositional registration (ie 0.6%), or even to below this value to as low as substantially 0.4%. This will further restrict the strengthening attributed to S' precipitation and will increase the solubility limit of lithium in aluminium thereby restricting the degree of δ' precipitation. Similarly, the lithium level may also need to be

maintained at or even below the 8090 compositional minimum (ie 2.2%). Reducing the copper levels may be counterproductive in terms of toughness and so further dilution below the Batch 2 level may not be advisable.

To further illustrate the benefit of reducing the ageing temperature according to the invention in order to increase the volume fraction of the δ' precipitate some recrystallised 8090 T31 sheet was aged for 24 hours at 170°C in order to reach a medium strength condition and then subsequently aged for 8 hours at 120°C. The longitudinal tensile properties after ageing for 24 hours at 170°C according to the prior art are shown below together with the properties after the subsequent 8 hours period of ageing at 120°C according to the invention. It can be seen that a significant increase in strength results from the inclusion of the relatively short ageing step at the lower temperature and that the final strength level attained is significantly higher than would have resulted from, say, 32 hours (ie 24 + 8 hours) at 170°C.

Ageing Treatment	0.2% Proof Stress (MPa)	Tensile Strength (MPa)	% Elongation
24 hrs @ 170°C	374	468	7
24 hrs @ 170°C			

+	406	499	8
---	-----	-----	---

8 hrs @ 120°C

The concept of RS-W ageing according to the invention to combine a prior art ageing step with a further ageing step or steps at reduced temperature to the initial ageing step to achieve a medium-to-high strength condition can therefore be seen to be advantageous in terms of maximising the strength that can be ultimately attained as well as achieving a given strength level in a shorter overall ageing time than would otherwise be possible. This type of processing is applicable to all Al-Li alloys strengthened, in part, by the precipitation of δ' and is applicable to all product forms such as plate, extrusions, forgings, tube etc. This particular form of the ageing treatment according to the invention is now termed High Strength Retrogressive Step-Wise ageing ("HSRS-W").

RANGE OF HEAT TREATMENTS

The nature of the heat treatment according to the RS-W aspect of the invention is such that there is a broad range of treatments which achieve approximately the same final condition. A very broad range of RS-W treatment intended to produce a condition of high plane stress fracture toughness is therefore disclosed and then various refinements culminating in a preferred range (RS-W Range

4) which is particularly suited to the 8090 alloy and which achieves an optimum combination of initial strength, toughness and thermal stability is disclosed.

The HSRS-W ageing treatment according to the invention combines the process of maximising the δ' volume fraction with an ageing treatment intended to produce a medium-to-high strength condition (ie high in S' and δ') to result in an increased strength level which is higher than would result from the initial prior art ageing treatment alone or from an isothermal ageing treatment of the same overall length which is solely carried out at the higher temperature.

For "short" ageing steps (ie less than or equal to substantially 3 hours) the time indicated may commence when the temperature of the product as determined by a contact-based temperature measuring device (thermocouple) reaches a temperature within 5°C of the nominal temperature of the treatment. Typically, for a 150°C ageing step applied to 1.6mm thick sheet and with the sheets loaded into a pre-heated air circulation oven, a heat up time of 10 to 15 minutes has been found to be appropriate.

For ageing times longer than about 3 hours the lag between the metal and oven air temperatures can be ignored and the treatment time then commences when the oven air temperature recovers to the set temperature.

For very short ageing treatments the use of an oil bath or similar may be necessary in place of an air oven. In such cases appropriate adjustments to the metal heat up times will be needed.

Treatments below 90°C are considered to be ineffective, according to the invention.

A continuous transition between the temperatures shown in any pair of adjoining steps is considered as part of the temperature ranges and time ranges specified.

RS-W TREATMENT - RANGE 1

	Temperature Range	Time Range
Step 1	165 to 130°C	15 Minutes to 24 Hours
Step 2	130 to 90°C	1 Hour to 72 Hours

RS-W TREATMENT - RANGE 2

	Temperature Range	Time Range
Step 1	160°C to 130°C	30 Minutes to 12 Hours
Step 2	130°C to 90°C	2 Hours to 72 Hours

RS-W TREATMENT - RANGE 3

	Temperature Range	Time Range
Step 1	150 ± 5°C	45 Minutes to 75 Minutes
Step 2	120 ± 5°C	4 to 12 Hours
Step 3	105 ± 5°C	12 to 36 Hours

Step 4 95 ± 5°C Zero to 24 Hours

RS-W TREATMENT - RANGE 4

	Temperature Range	Time Range
Step 1	150 ± 5°C	1 Hour ± 15 Minutes
Step 2	120 ± 5°C	8 ± 2 Hours
Step 3	105 ± 5°C	24 ± 6 Hours
Step 4	95 ± 5°C	Zero to 8 Hours

HSRS-W

The HSRS-W treatment ranges are described either as 2-step or as 3/4-step (ie 4-step treatment but with the fourth step optional which, if omitted, thereby results in a 3-step treatment).

HSRS-W TREATMENT - 2-STEP, RANGE 1

	Temperature Range	Time Range
Step 1	190 ± 40°C	20 Minutes to 72 Hours
Step 2	120 ± 30°C	1 Hour to 48 Hours

HSRS-W TREATMENT - 2-STEP, RANGE 2

	Temperature Range	Time Range
Step 1	170 ± 20°C	4 Hours to 48 Hours
Step 2	125 ± 15°C	4 Hours to 36 Hours

HSRS-W TREATMENT - 2-STEP, RANGE 3

	Temperature Range	Time Range
Step 1	170 \pm 20°C	12 Hours to 36 Hours
Step 2	125 \pm 15°C	6 Hours to 24 Hours

HSRS-W TREATMENT - 2-STEP, RANGE 4

	Temperature Range	Time Range
Step 1	170 \pm 10°C	24 \pm 4 Hours
Step 2	125 \pm 10°C	8 \pm 2 Hours

HSRS-W TREATMENT - 3/4 STEP, RANGE 1

	Temperature Range	Time Range
Step 1	170 \pm 20°C	4 Hours to 48 Hours
Step 2	125 \pm 15°C	6 Hours to 24 Hours
Step 3	105 \pm 10°C	8 Hours to 30 Hours
Step 4	95 \pm 5°C	Zero to 8 Hours

HSRS-W TREATMENT - 3/4-STEP, RANGE 2

	Temperature Range	Time Range
Step 1	170 \pm 10°C	24 \pm 4 Hours
Step 2	125 \pm 10°C	8 \pm 4 Hours
Step 3	105 \pm 5°C	18 \pm 6 Hours
Step 4	95 \pm 5°C	Zero to 8 Hours

In summary the use of the RS-W ageing method of the invention provides a means of achieving a strength level for aluminium-lithium alloys such as 8090 which are

strengthened by the precipitation of δ' and S' which is comparable with conventional aluminium-copper alloy materials whilst also restricting the degree of subsequent and unwanted strengthening and associated loss in fracture toughness which can take place due to prolonged exposure to moderately elevated temperatures such as are encountered by fuselage, wing and empennage skin structures during on-the-ground exposures when relatively high ambient temperatures exist and/or there is significant heating due to solar radiation.

The use of the HSR-S-W ageing method of the invention provides a means of achieving a strength level for aluminium-lithium alloys such as 8090 which are strengthened by the precipitation of δ' and S' which is comparable with conventional aluminium-copper and also aluminium-zinc alloy materials

The invention also provides a means of achieving an improved level of toughness of all other aluminium-lithium alloys whether in plate form, sheet form, extruded form or otherwise primarily strengthened by the precipitation of the δ' (Al_3Li) precipitate in conjunction with other precipitates such as S' (Al_2CuMg).

In addition the invention also provides an improvement in the resistance of the 8090 alloy in recrystallised sheet form to intergranular corrosion.

INITIAL CONDITION	AS-RECEIVED PROPERTIES				PROPERTIES AFTER 920 HOURS @ 100°C			
	0.2% PROOF STRESS MPa	TENSILE STRENGTH MPa	ELONG. %	ELECT. COND. XIACS	0.2% PROOF STRESS MPa	TENSILE STRENGTH MPa	ELONG. %	ELECT. COND. XIACS
T81 (T31 + 150°C/24 hours)	293	424	13.5	18.8	320	439	10.2	19.6
T81 + REVERSION (200°C/10 MINUTES)	260	379	14.8	17.6	324	451	10.5	19.8
T81 + 200°C/10 MINUTES + 170°C/4 HOURS	295	416	13.6	18.6	339	471	10.0	20.5
T81 + 220°C/12 HOURS	346	411	8.4	18.5	394	471	5.4	20.4

TABLE 1 ROOM TEMPERATURE MECHANICAL PROPERTIES AND ELECTRICAL CONDUCTIVITY OF BATCH 1 8090 IN VARIOUS INITIAL CONDITIONS AFTER 920 HOURS AT 100°C THERMAL EXPOSURE.

AGEING TREATMENT			THERMAL		PROOF STRESS		TENSILE	ELONGATION	ROOM TEMP. ELECTRICAL
(HOURS AT TEMPERATURE)			SENSITISATION		0.1X	0.2X	STRENGTH	%	CONDUCTIVITY
150°C	135°C	120°C	85°C	70°C	MPa	MPa	MPa		KLACS
1	-	-	-	-	205	216	342	20.0	17.5
1	6	-	-	-	249	260	384	15.8	18.2
1	6	3	-	-	256	267	392	15.8	18.6
1	6	3	50	-	255	277	408	15.8	19.0
1	6	3	50	100	274	285	413	13.9	19.3
1	6	3	50	500	282	294	416	14.3	19.4
1	6	3	50	500	284	294	416	13.5	19.6

27

TABLE 2A

LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 1 1.6 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 1h/150°C + 6h/135°C + 3h/120°C + 50h/100°C AND AFTER THERMAL EXPOSURE AT 85°C & 70°C.

STARTING CONDITION: SOLUTION TREATED 505°C AND CONTROLLED STRETCHED $2\% \pm 0.5\%$ IN LONGITUDINAL DIRECTION.

AGEING TREATMENT				THERMAL		PROOF STRESS		TENSILE	ELONGATION	ROOM TEMP. ELECTRICAL
(HOURS AT TEMPERATURE)				SENSITISATION		0.1%	0.2%	STRENGTH	%	CONDUCTIVITY
150°C	135°C	120°C	100°C	85°C	70°C	MPa	MPa	MPa		XIACS
1	-	-	-	-	-	205	216	342	20.0	17.5
1	6	-	-	-	-	249	260	384	15.8	18.2
1	6	8	-	-	-	252	269	393	14.9	18.6
1	6	8	50	-	-	264	280	406	14.5	19.0
1	6	8	50	100	-	277	287	415	14.5	19.3
1	6	8	50	500	-	284	296	426	16.1	19.3
1	6	8	50	500	500	281	292	419	13.6	19.6

28

TABLE 2B LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 1 1.6 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 1h/150°C + 6h/135°C + 8h/120°C + 50h/100°C AND AFTER THERMAL EXPOSURE AT 85°C & 70°C.

STARTING CONDITION: SOLUTION TREATED 505°C AND CONTROLLED STRETCHED $2\% \pm 0.5\%$ IN LONGITUDINAL DIRECTION.

AGEING TREATMENT			THERMAL		PROOF STRESS			TENSILE	ELONGATION	ROOM TEMP. ELECTRICAL	
(HOURS AT TEMPERATURE)			SENSITISATION		0.1%	0.2%	0.5%	STRENGTH	%	CONDUCTIVITY	
150°C	135°C	120°C	85°C	70°C	MPa	MPa	MPa	MPa		XIACS	
1	-	-	-	-	205	216	238	342	20.0	17.5	
1	6	-	-	-	249	260	284	384	15.8	18.2	
1	6	16	-	-	265	275	301	403	15.4	18.7	
1	6	16	-	-	251	280	306	407	15.8	19.1	
1	6	16	100	-	276	287	312	413	14.9	19.3	29
1	6	16	500	-	283	295	320	425	13.3	19.4	
1	6	16	500	500	283	294	319	420	12.2	19.7	

TABLE 2C

LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 1 1.6 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 1h/150°C + 6h/135°C + 16h/120°C + 50h/100°C AND AFTER THERMAL EXPOSURE AT 85°C, 70°C.

STARTING CONDITION: SOLUTION TREATED 505°C AND CONTROLLED STRETCHED $2\% \pm 0.5\%$ IN LONGITUDINAL DIRECTION.

AGEING TREATMENT				THERMAL		PROOF STRESS		TENSILE	ELONGATION	ROOM TEMP. ELECTRICAL
(HOURS AT TEMPERATURE)				SENSITISATION		0.1%	0.2%	STRENGTH	%	CONDUCTIVITY
150°C	135°C	120°C	100°C	85°C	70°C	MPa	MPa	MPa		ZIACS
1	-	-	-	-	-	205	216	238	20.0	17.5
1	12	-	-	-	-	260	270	295	14.7	18.5
1	12	6	-	-	-	269	278	302	14.7	18.8
1	12	6	50	-	-	272	287	312	14.1	19.1
1	12	6	50	100	-	274	290	316	14.8	19.4
1	12	6	50	500	-	292	301	325	16.8	19.5
1	12	6	50	500	500	289	300	325	13.3	19.7

30

TABLE 2D LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 1 1.6 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 1h/150°C + 12h/135°C/ + 6h/120°C + 50h/100°C AND AFTER THERMAL EXPOSURE AT 85°C & 70°C.

STARTING CONDITION: SOLUTION TREATED 505°C AND CONTROLLED STRETCHED $2\% \pm 0.5\%$ IN LONGITUDINAL DIRECTION.

AGEING TREATMENT				THERMAL		PROOF STRESS			TENSILE	ELONGATION	ROOM TEMP. ELECTRICAL
(HOURS AT TEMPERATURE)				SENSITISATION		0.1%	0.2%	0.5%	STRENGTH	%	CONDUCTIVITY
150°C	135°C	120°C	100°C	85°C	70°C	MPa	MPa	MPa	MPa		XIACS
1	-	-	-	-	-	205	216	238	342	20.0	17.5
1	12	-	-	-	-	260	270	295	393	14.7	18.5
1	12	16	-	-	-	274	284	309	410	15.5	18.9
1	12	16	50	-	-	274	289	314	417	13.6	19.2
1	12	16	50	100	-	283	295	319	422	12.8	19.5
1	12	16	50	500	-	290	299	324	427	11.8	19.6
1	12	16	50	500	500	292	302	327	427	12.5	19.8

31

TABLE 2B

LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 1 1.6 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 1h/150°C + 12h/135°C + 16h/120°C + 50h/100°C AND AFTER THERMAL EXPOSURE AT 85°C & 70°C.

STARTING CONDITION: SOLUTION TREATED 505°C AND CONTROLLED STRETCHED $2\% \pm 0.5\%$ IN LONGITUDINAL DIRECTION.

AGEING TREATMENT (HOURS AT TEMPERATURE)				THERMAL		PROOF STRESS			TENSILE	ELONGATION	ROOM TEMP. ELECTRICAL	
150°C	135°C	120°C	100°C	SENSITISATION	85°C	70°C	0.1% MPa	0.2% MPa	0.5% MPa	STRENGTH MPa	%	CONDUCTIVITY XIACS
3	-	-	-	-	-	-	237	247	270	372	16.0	17.9
3	12	-	-	-	-	-	266	279	304	406	15.0	18.7
3	12	6	-	-	-	-	277	287	311	415	17.4	19.0
3	12	6	50	-	-	-	264	293	318	421	14.3	19.3
3	12	6	50	100	-	-	285	296	322	423	13.3	19.6
3	12	6	50	500	-	-	291	301	325	429	13.6	19.7
3	12	6	50	500	500	500	291	302	326	429	14.1	19.9

32

TABLE 2F

LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 1 1.6 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 3h/150°C + 12h/135°C + 6h/120°C + 50h/100°C AND AFTER THERMAL EXPOSURE AT 85°C & 70°C.

STARTING CONDITION: SOLUTION TREATED 505°C AND CONTROLLED STRETCHED $2\% \pm 0.5\%$ IN LONGITUDINAL DIRECTION.

AGEING TREATMENT (HOURS AT TEMPERATURE)			THERMAL SENSITISATION		PROOF STRESS			TENSILE STRENGTH MPa	ELONGATION %	ROOM TEMP. ELECTRICAL CONDUCTIVITY XIACS	
150°C	135°C	120°C	100°C	85°C	70°C	0.1% MPa	0.2% MPa	0.5% MPa			
3	-	-	-	-	-	237	247	270	372	16.0	17.9
3	12	-	-	-	-	266	279	304	406	15.0	18.7
3	12	16	-	-	-	280	291	316	422	16.5	19.1
3	12	16	50	-	-	275	291	317	418	13.3	19.4
3	12	16	50	100	-	279	298	324	426	12.3	19.6
3	12	16	50	500	-	294	303	328	434	12.6	19.7
3	12	16	50	500	500	294	306	331	436	11.8	20.0

33

TABLE 2G

LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 1 1.6 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 3h/150°C + 12h/135°C + 16h/120°C + 50h/100°C AND AFTER THERMAL EXPOSURE AT 85°C & 70°C.

STARTING CONDITION: SOLUTION TREATED 505°C AND CONTROLLED STRETCHED $2\% \pm 0.5\%$ IN LONGITUDINAL DIRECTION.

AGEING TREATMENT			THERMAL		PROOF STRESS			TENSILE	ELONGATION	ROOM TEMP. ELECTRICAL	
(HOURS AT TEMPERATURE)			SENSITISATION		0.1% MPa	0.2% MPa	0.5% MPa	STRENGTH MPa	%	CONDUCTIVITY %IACS	
150°C	135°C	120°C	100°C	85°C	70°C						
1	-	-	-	-	-	224.2	232.0	254.3	366.3	20.6	16.4
1	6	-	-	-	-	259.1	267.3	290.8	398.2	18.5	17.5
1	6	8	-	-	-	275.4	283.4	307.9	414.3	14.4	17.9
1	6	8	50	-	-	287.2	295.1	320.2	430.0	16.8	18.3
1	6	8	50	100	-	288.7	296.5	320.9	429.8	17.2	18.5
1	6	8	50	250	-	290.5	298.0	322.1	429.3	14.6	18.6
1	6	8	50	250	500	297.3	309.7	328.3	434.5	12.7	18.8
1	6	8	50	-	-	(301.7)	(307.3)	(320.6)	(415.2)	(12.8)	(18.3)

34

TABLE 3A

LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 2 1.9 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 1h/150°C + 6h/135°C + 8h/120°C + 50h/100°C AND AFTER THERMAL EXPOSURE AT 85°C & 70°C. (LONGITUDINAL RESULTS SHOWN IN PARENTHESIS).

STARTING CONDITION: SOLUTION TREATED 530°C AND CONTROLLED STRETCHED 2% + 0.5% IN LONGITUDINAL DIRECTION.

AGEING TREATMENT (HOURS AT TEMPERATURE)			THERMAL		PROOF STRESS			TENSILE	ELONGATION	ROOM TEMP. ELECTRICAL	
150°C	120°C	105°C	95°C	85°C	70°C	0.1% MPa	0.2% MPa	0.5% MPa	STRENGTH MPa	%	CONDUCTIVITY XIACS
1	-	-	-	-	-	224.2	232.0	254.3	366.3	20.6	16.4
1	8	-	-	-	-	253.7	260.9	283.3	394.3	18.3	17.4
1	8	24	-	-	-	268.1	275.5	299.2	409.7	18.0	17.8
1	8	24	24	-	-	274.1	281.8	306.2	413.6	19.1	17.9
1	8	24	24	100	-	277.4	284.7	308.4	416.3	14.7	18.2
1	8	24	24	250	-	283.2	291.0	315.8	422.4	17.7	18.3
1	8	24	24	250	500	288.5	296.1	320.5	427.0	16.8	18.4
1	8	24	24	250	500	287.9	294.7	317.7	426.9	19.5	18.4
1	8	24	24	-	-	(288.7)	(293.5)	(305.9)	(402.5)	(13.9)	(17.9)

35

TABLE 3B LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 2 1.9 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 1h/150°C + 8h/120°C + 24h/105°C + 24h/95°C AND AFTER THERMAL EXPOSURE AT 85°C, 70°C. (LONGITUDINAL RESULTS SHOWN IN PARENTHESES).

STARTING CONDITION: SOLUTION TREATED 530°C AND CONTROLLED STRETCHED 2X ± 0.5% IN LONGITUDINAL DIRECTION.

AGEING TREATMENT (HOURS AT TEMPERATURE)				THERMAL SENSITISATION		PROOF STRESS			TENSILE STRENGTH	ELONGATION	ROOM TEMP. ELECTRICAL CONDUCTIVITY
150°C	120°C	105°C	95°C	85°C	70°C	0.1%	0.2%	0.5%	MPa	%	XIACS
1	-	-	-	-	-	224.2	232.0	254.3	366.3	20.6	16.4
1	16	-	-	-	-	264.4	272.1	295.1	405.7	18.5	17.5
1	16	24	-	-	-	274.1	281.9	305.7	415.6	19.0	18.0
1	16	24	24	-	-	276.9	284.5	309.1	419.7	16.4	18.1
1	16	24	24	100	-	274.8	282.5	306.1	417.7	17.6	18.3
1	16	24	24	250	-	285.8	293.6	317.9	424.5	14.4	18.4
1	16	24	24	250	500	290.7	298.4	323.2	433.6	17.6	18.6
1	16	24	24	-	-	(299.4)	(304.7)	(316.3)	(405.7)	(12.6)	(18.1)

36

TABLE 3C

LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 2 1.9 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 1h/150°C + 16h/120°C + 24h/105°C + 24h/95°C AND AFTER THERMAL EXPOSURE AT 85°C, 70°C. (LONGITUDINAL RESULTS SHOWN IN PARENTHESIS).

STARTING CONDITION: SOLUTION TREATED 530°C AND CONTROLLED STRETCHED $2\% \pm 0.5\%$ IN LONGITUDINAL DIRECTION.

AGING TREATMENT				THERMAL			PROOF STRESS			TENSILE	ELONGATION	ROOM TEMP. ELECTRICAL
(HOURS AT TEMPERATURE)				SENSITISATION			0.1%	0.2%	0.5%	STRENGTH	%	CONDUCTIVITY
150°C	125°C	105°C	95°C	85°C	70°C		MPa	MPa	MPa	MPa		XIACS
1	-	-	-	-	-	-	224.2	232.0	254.3	366.3	20.6	16.4
1	8	-	-	-	-	-	254.5	263.2	286.4	398.2	18.7	17.4
1	8	24	-	-	-	-	269.8	277.7	300.7	410.8	14.1	17.9
1	8	24	24	-	-	-	275.6	282.9	306.6	417.4	17.7	18.1
1	8	24	24	100	-	-	282.0	289.3	312.3	423.8	17.1	18.3
1	8	24	24	250	-	-	286.6	294.1	318.0	428.3	16.5	18.4
1	8	24	24	250	500	-	287.3	294.8	318.7	424.9	17.1	18.5
1	8	24	24	250	500	-	286.0	293.1	316.5	424.6	16.3	18.5
1	8	24	24	-	-	-	(293.7)	(299.6)	(312.1)	(403.2)	(12.7)	(18.0)

TABLE 3D LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 2 1.9 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 1h/150°C + 8h/125°C + 24h/105°C + 24h/95°C AND AFTER THERMAL EXPOSURE AT 85°C, 70°C. (LONGITUDINAL RESULTS SHOWN IN PARENTHESES).

STARTING CONDITION: SOLUTION TREATED 530°C AND CONTROLLED STRETCHED $2\% \pm 0.5\%$ IN LONGITUDINAL DIRECTION.

AGEING TREATMENT (HOURS AT TEMPERATURE)			THERMAL		PROOF STRESS		TENSILE	ELONGATION	ROOM TEMP. ELECTRICAL
150°C	125°C	105°C	95°C	SENSITISATION 85°C	0.1% MPa	0.2% MPa	0.5% MPa	%	CONDUCTIVITY XIACS
1	-	-	-	-	224.2	232.0	254.3	20.6	16.4
1	16	-	-	-	267.1	274.9	298.8	17.6	17.6
1	16	24	-	-	279.6	287.4	311.6	20.1	18.1
1	16	24	24	-	285.1	292.7	317.0	14.9	18.2
1	16	24	24	100	287.9	295.4	319.2	14.8	18.4
1	16	24	24	250	291.5	299.4	324.7	15.9	18.5
1	16	24	24	250	293.2	300.5	324.0	15.8	18.7
1	16	24	24	-	(301.4)	(306.8)	(318.7)	(12.4)	(18.2)

38

TABLE 3E

LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 2 1.9 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 1h/150°C + 16h/125°C + 24h/105°C + 24h/95°C AND AFTER THERMAL EXPOSURE AT 85°C, 70°C. (LONGITUDINAL RESULTS SHOWN IN PARENTHESIS).

STARTING CONDITION: SOLUTION TREATED 530°C AND CONTROLLED STRETCHED $2\% \pm 0.5\%$ IN LONGITUDINAL DIRECTION.

AGEING TREATMENT (HOURS AT TEMPERATURE)				THERMAL		PROOF STRESS			TENSILE	ELONGATION	ROOM TEMP. ELECTRICAL
135°C	120°C	105°C	95°C	85°C	70°C	0.1% MPa	0.2% MPa	0.5% MPa	STRENGTH MPa	%	CONDUCTIVITY XIACS
1	-	-	-	-	-	198.4	205.9	225.8	341.6	22.4	15.9
1	8	-	-	-	-	232.2	239.4	260.6	374.4	19.3	16.8
1	8	24	-	-	-	252.1	259.5	282.1	399.3	20.3	17.4
1	8	24	24	-	-	256.6	264.2	286.5	399.0	20.3	17.5
1	8	24	24	100	-	267.3	274.9	298.3	412.8	19.5	17.9
1	8	24	24	250	-	278.2	285.6	309.3	418.3	15.5	18.0
1	8	24	24	250	500	279.4	286.6	309.4	420.3	16.3	18.2
1	8	24	24	250	1250	283.8	290.5	313.0	425.4	17.2	18.2
1	8	24	24	-	-	(273.9)	(278.3)	(290.8)	(386.9)	(10.5)	(17.5)

TABLE 3F

LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 2 1.9 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 1h/135°C + 8h/120°C + 24h/105°C + 24h/95°C AND AFTER THERMAL EXPOSURE AT 85°C & 70°C. (LONGITUDINAL RESULTS SHOWN IN PARENTHESIS).

STARTING CONDITION: SOLUTION TREATED 530°C AND CONTROLLED STRETCHED $2\% \pm 0.5\%$ IN LONGITUDINAL DIRECTION.

AGEING TREATMENT			THERMAL		PROOF STRESS			TENSILE	ELONGATION	ROOM TEMP. ELECTRICAL	
(HOURS AT TEMPERATURE)			SENSITISATION		0.1%	0.2%	0.5%	STRENGTH	%	CONDUCTIVITY	
135°C	120°C	105°C	85°C	70°C	MPa	MPa	MPa	MPa		XIACS	
1	-	-	-	-	198.4	205.9	225.8	341.6	22.4	15.9	
1	16	-	-	-	245.3	252.7	274.8	387.5	22.8	17.2	
1	16	24	-	-	258.9	266.2	288.8	400.0	19.0	17.5	
1	16	24	-	-	261.8	269.6	292.5	395.5	16.4	17.8	
1	16	24	100	-	270.2	277.2	299.5	414.8	18.1	18.0	
1	16	24	250	-	280.2	287.9	311.9	420.6	15.9	18.1	
1	16	24	250	500	282.4	288.9	311.6	417.6	16.7	18.3	
1	16	24	250	1250	289.2	296.5	319.7	425.8	14.9	18.4	
1	16	24	-	-	(286.6)	(292.0)	(303.8)	(399.5)	(11.8)	(17.8)	

40

TABLE 3C LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 2 1.9 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 1h/135°C + 16h/120°C + 24h/105°C + 24h/95°C AND AFTER THERMAL EXPOSURE AT 85°C, 70°C. (LONGITUDINAL RESULTS SHOWN IN PARENTHESES).

STARTING CONDITION: SOLUTION TREATED 530°C AND CONTROLLED STRETCHED 2% ± 0.5% IN LONGITUDINAL DIRECTION.

AGEING TREATMENT (HOURS AT TEMPERATURE)			THERMAL SENSITISATION		PROOF STRESS			TENSILE STRENGTH	ELONGATION	ROOM TEMP. ELECTRICAL CONDUCTIVITY
135°C	120°C	105°C	85°C	70°C	0.1X	0.2X	0.5X	MPa	%	XIACS
-	2	-	-	-	189.5	196.2	213.5	336.1	20.7	15.7
-	2	32	-	-	235.2	242.2	263.5	375.5	21.4	16.8
-	2	32	-	-	242.7	249.9	271.3	386.7	18.6	17.1
-	2	32	100	-	256.2	263.6	286.2	403.7	19.3	17.5
-	2	32	250	-	267.7	274.9	297.2	411.9	16.9	17.7
-	2	32	250	500	272.4	279.2	301.3	414.3	15.8	18.0
-	2	32	250	1250	276.1	283.5	306.5	412.5	17.1	18.0
-	2	32	-	-	(260.0)	(263.8)	(274.8)	(377.4)	(16.6)	(17.1)

TABLE 3H

LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 2 1.9 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGING SEQUENCE 2h/120°C + 32h/120°C + 24h/95°C AND AFTER THERMAL EXPOSURE AT 85°C & 70°C. (LONGITUDINAL RESULTS SHOWN IN PARENTHESIS).

STARTING CONDITION: SOLUTION TREATED 530°C AND CONTROLLED STRETCHED $2\% \pm 0.5\%$ IN LONGITUDINAL DIRECTION.

AGEING TREATMENT			THERMAL		PROOF STRESS			TENSILE	ELONGATION	ROOM TEMP. ELECTRICAL	
(HOURS AT TEMPERATURE)			SENSITISATION		0.1% MPa	0.2% MPa	0.5% MPa	STRENGTH MPa	%	CONDUCTIVITY %IACS	
135°C	120°C	105°C	85°C	70°C							
-	8	-	-	-	217.8	224.9	244.8	364.1	21.5		16.4
-	8	24	-	-	240.6	247.5	268.4	389.9	18.6		17.1
-	8	24	-	-	249.5	256.7	279.1	388.7	18.3		17.4
-	8	24	100	-	262.6	269.6	291.0	408.8	16.5		17.6
-	8	24	250	-	271.9	278.6	300.9	415.9	19.1		17.8
-	8	24	250	500	271.3	278.6	300.7	413.1	20.5		18.1
-	8	24	250	1250	279.0	286.0	308.7	416.4	17.0		18.1
-	8	24	-	-	(265.2)	(269.8)	(281.1)	(384.1)	(167.6)		(17.3)

TABLE 3J LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 2 1.9 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 8h/120°C + 24h/120°C + 24h/95°C AND AFTER THERMAL EXPOSURE AT 85°C & 70°C. (LONGITUDINAL RESULTS SHOWN IN PARENTHESES).

STARTING CONDITION: SOLUTION TREATED 530°C AND CONTROLLED STRETCHED $2\% \pm 0.5\%$ IN LONGITUDINAL DIRECTION.

AGEING TREATMENT (HOURS AT TEMPERATURE)				THERMAL		PROOF STRESS		TENSILE	ELONGATION	ROOM TEMP. ELECTRICAL
150°C	120°C	105°C	95°C	85°C	70°C	0.1% MPa	0.2% MPa	STRENGTH MPa	%	CONDUCTIVITY %IACS
0.75	8	-	-	-	-	241.6	248.7	389.4	20.7	17.6
0.75	8	24	-	-	-	261.6	268.4	405.2	20.1	18.0
0.75	8	24	8	-	-	262.5	270.2	406.3	18.6	18.2
0.75	8	24	24	-	-	268.3	276.1	417.5	19.6	18.2

43

TABLE 4A LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 2 1.6 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 0.75h/150°C + 8h/120°C + 24h/105°C + 8h/95°C OR 24h/95°C.

STARTING CONDITION: SOLUTION TREATED 530°C AND CONTROLLED STRETCHED 1.75% ± 0.25% IN LONGITUDINAL DIRECTION.

AGEING TREATMENT (HOURS AT TEMPERATURE)				THERMAL SENSITISATION			PROOF STRESS			TENSILE STRENGTH	ELONGATION	ROOM TEMP. ELECTRICAL CONDUCTIVITY
150°C	120°C	105°C	95°C	85°C	70°C	0.1% MPa	0.2% MPa	0.5% MPa	MPa	MPa	%	μIACS
1.00	8	-	-	-	-	250.1	258.2	283.4	394.4	18.3	17.8	
1.00	8	24	-	-	-	266.7	274.8	299.7	411.3	19.3	18.1	
1.00	8	24	8	-	-	272.1	280.2	305.8	421.0	18.1	18.3	
1.00	8	24	24	-	-	273.6	281.5	306.3	415.8	16.2	18.3	44

TABLE 4B

LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 2 1.6 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 1h/150°C + 8h/120°C + 24h/105°C + 8h/95°C OR 24h/95°C.

STARTING CONDITION: SOLUTION TREATED 530°C AND CONTROLLED STRETCHED $1.75\% \pm 0.25\%$ IN LONGITUDINAL DIRECTION.

AGEING TREATMENT			THERMAL		PROOF STRESS			TENSILE	ELONGATION	ROOM TEMP. ELECTRICAL
(HOURS AT TEMPERATURE)			SENSITISATION		0.1% MPa	0.2% MPa	0.5% MPa	STRENGTH MPa	%	CONDUCTIVITY XIACS
150°C	120°C	105°C	85°C	70°C						
1.25	8	-	-	-	247.8	255.1	278.6	391.4	18.9	17.9
1.25	8	24	-	-	270.7	278.9	304.5	415.2	16.8	18.2
1.25	8	24	8	-	272.9	280.9	306.2	419.7	16.8	18.3
1.25	8	24	24	-	272.2	279.4	303.4	416.8	18.0	18.4

45

TABLE 4C LONG TRANSVERSE TENSILE PROPERTIES AND ELECTRICAL CONDUCTIVITY MEASUREMENTS FOR BATCH 2 1.6 mm 8090 SHEET AT EACH STAGE OF AGEING FOR AGEING SEQUENCE 1.25h/150°C + 8h/120°C + 24h/105°C + 8h/95°C OR 24h/95°C.

STARTING CONDITION: SOLUTION TREATED 530°C AND CONTROLLED STRETCHED 1.75% ± 0.25% IN LONGITUDINAL DIRECTION.

THERMAL EXPOSURE HOURS @ 70°C	STARTING CONDITION	0.2% PROOF STRESS MPa	TENSILE STRENGTH MPa	ELONGATION %
- (CONTROL)	T81	309.4 ¹	441.3 ¹	13.3 ¹
- (CONTROL)	RS-W	279.0 ²	413.7 ²	16.6 ²
100	T81	314.5	449.4	13.9
100	RS-W	284.9 ¹	416.7 ¹	16.8 ¹
200	T81	315.5	446.1	14.2
200	RS-W	286.7 ¹	422.3 ¹	17.3 ¹
500	T81	314.2	451.9	13.3
500	RS-W	291.2 ¹	431.7 ¹	15.8 ¹
1000	T81	316.4	454.3	11.1
1000	RS-W	297.7 ¹	440.4 ¹	16.1 ¹
2000	T81	330.7 ¹	466.3 ¹	12.6 ¹
2000	RS-W	300.8 ¹	436.9 ¹	15.7 ¹

TABLE 5 ROOM TEMPERATURE LONG TRANSVERSE TENSILE PROPERTIES FOR BATCH 2 1.6 mm 8090 SHEET 70°C THERMAL EXPOSURE TRIAL INVOLVING T81 AND MATERIAL AGED TO THE PREFERRED RS-W CONDITION (i.e. 1h/150°C + 8h/120°C + 24h/105°C + 8h/95°C).

¹ Average of 2 tests.

² Average of 16 tests. The extreme highest and lowest values of 0.2% Proof Stress for the RS-W "Control" tests were 2.3 MPa above the mean and 2.5 MPa below the mean.

CLAIMS

1. A method of heat treating an aluminium-lithium alloy including carrying out a succession of at least two artificial ageing steps, the first such step being carried out within a first temperature range and at least one further step being carried out within a successively reduced temperature range.
2. A method as in Claim 1 including carrying out the first artificial ageing step substantially within the temperature range 165°C to 130°C and substantially within a time range of 15 minutes to 24 hours and thereafter carrying out a second said artificial ageing step substantially within a temperature range 130°C to 90°C and substantially within a time range of 1 hour to 72 hours.
3. A method as in Claim 1 including carrying out the first artificial ageing step substantially within a temperature range 160°C to 130°C and substantially within a time range of 30 minutes to 12 hours and thereafter carrying out a second artificial ageing step substantially within a temperature range 130°C to 90°C and substantially within a time range of 2 hours to 72 hours.
4. A method as in Claim 1 including carrying out the first artificial ageing step substantially within a time range of 155°C to 145°C and substantially within a time range of 45 minutes to 75 minutes, thereafter carrying out

a second artificial ageing step substantially within a temperature range 125°C to 115°C and substantially within a time range 4 hours to 12 hours, thereafter carrying out a third artificial ageing step substantially within a temperature range 110°C to 100°C and substantially within a time range 12 hours to 36 hours and thereafter carrying out a fourth artificial ageing step substantially within a temperature range 100°C to 90°C and substantially within a time range 0 hours to 24 hours.

5. A method as in Claim 1 including carrying out the first artificial ageing step substantially within a temperature range 155°C to 145°C and substantially within a time range 45 minutes to 75 minutes, thereafter carrying out a second artificial ageing step substantially within a temperature range 125°C to 115°C and substantially within a time range 6 hours to 10 hours, thereafter carrying out a third artificial ageing step substantially within a temperature range 110°C to 100°C and substantially within a time range 18 hours to 30 hours and thereafter carrying out a fourth artificial ageing step substantially within a temperature range 100°C to 90°C and substantially within a time range 0 hours to 8 hours.

6. A method as in Claim 1 including carrying out the first artificial ageing step substantially within a temperature range 230°C to 150°C and substantially within a time range 20 minutes to 72 hours and thereafter carrying

out a second artificial ageing step substantially within a temperature range 150°C to 90°C and substantially within a time range 1 hour to 48 hours.

7. A method as in Claim 1 including carrying out the first artificial ageing step substantially within a temperature range 190°C to 150°C and substantially within a time range 4 hours to 48 hours and thereafter carrying out a second artificial ageing step substantially within a temperature range 140°C to 110°C and substantially within a time range 4 hours to 36 hours.

8. A method as in Claim 1 including carrying out the first artificial ageing step substantially within a temperature range 190°C to 150°C and substantially within a time range 12 hours to 36 hours and thereafter carrying out a second artificial ageing step substantially within a temperature range 140°C to 110°C and substantially within a time range 6 hours to 24 hours.

9. A method as in Claim 1 including carrying out the first artificial ageing step substantially within a temperature range 180°C to 160°C and substantially within a time range 20 hours to 28 hours and thereafter carrying out a second artificial ageing step substantially within a temperature range 135°C to 115°C and substantially within a time range 6 hours to 10 hours.

10. A method as in Claim 1 including carrying out the first artificial ageing step substantially within a

temperature range 190°C to 150°C and substantially within a time range 4 hours to 48 hours, thereafter carrying out a second artificial ageing step substantially within a temperature range 140°C to 110°C and substantially within a time range 6 hours to 24 hours, thereafter carrying out a third artificial ageing step substantially within a temperature range 115°C to 95°C and substantially within a time range 8 hours to 30 hours and thereafter carrying out a fourth artificial ageing step substantially within a temperature range 100°C to 90°C and substantially within a time range 0 hours to 8 hours.

11. A method as in Claim 1 including carrying out the first artificial ageing step substantially within a temperature range 180°C to 160°C and substantially within a time range 20 hours to 28 hours and thereafter carrying out a second artificial ageing step substantially within a temperature range 135°C to 115°C and substantially within a time range 4 hours to 12 hours, thereafter carrying out a third artificial ageing step substantially within a temperature range 110°C to 100°C and substantially within a time range 12 hours to 24 hours and thereafter carrying out a fourth artificial ageing step substantially within a temperature range 100°C to 90°C and substantially within a time range 0 hours to 8 hours.

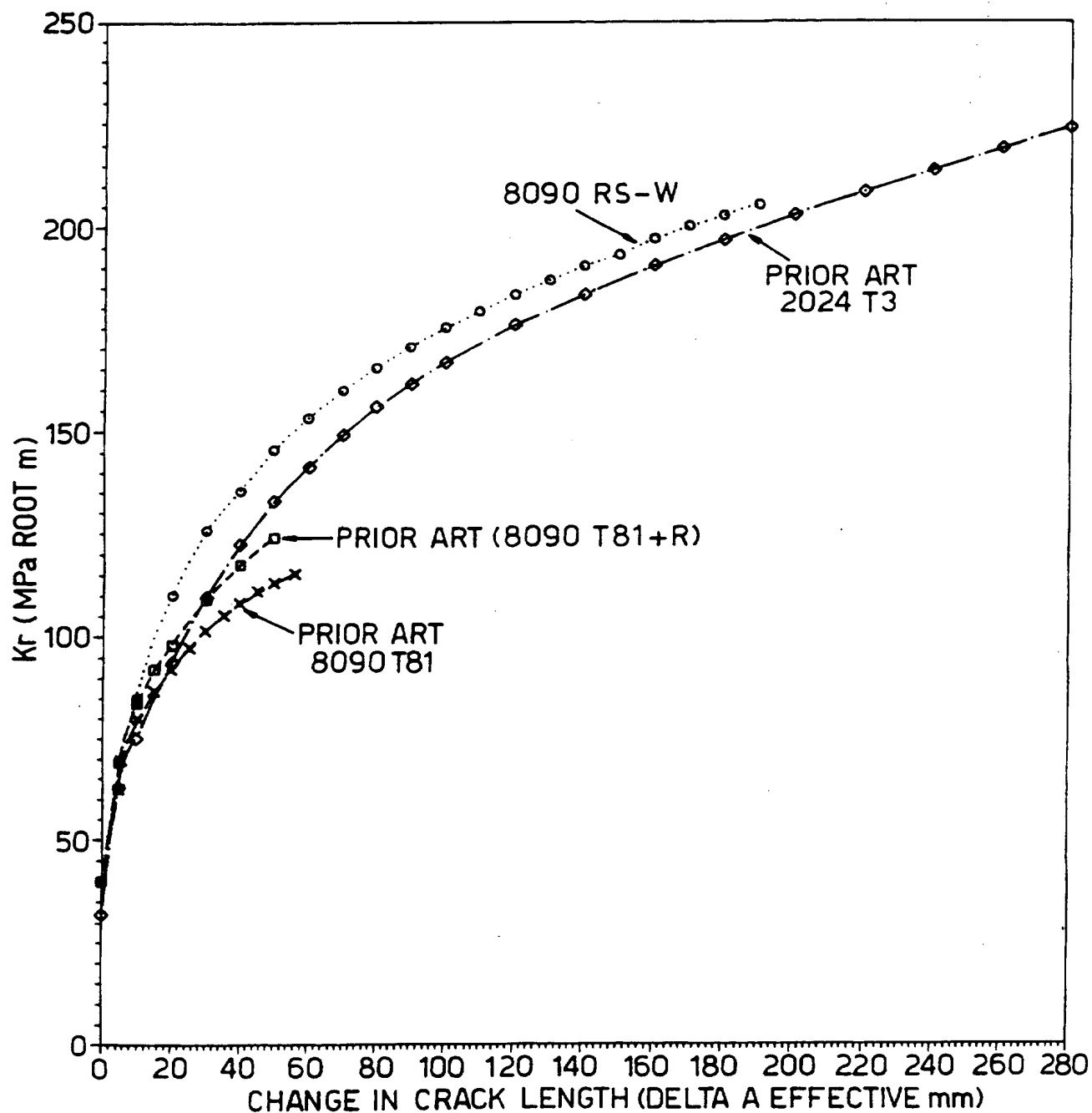
12. A method of forming an adhesively bonded heat treated structure of at least two components at least one

of which comprises aluminium-lithium alloy, the method including the steps of forming a pre-cure assembly of the components and adhesive and heat treating the assembly according to the method of Claim 1 whereby to cure the adhesive during at least one of the artificial ageing steps and so form the adhesively bonded heat treated structure.

13. A method of heat treating an aluminium-lithium alloy substantially as herein described.

14. A method of forming an adhesively bonded heat treated structure substantially as herein described.

Fig.1.



- 8090 RS-W BATCH 2-1.9mm (W=2000mm)
- ALCLAD 2024 T3-1.6mm REF 2 (W=200mm)
- 8090 T81+R BATCH L0346 REF.1 (W=760mm)
- x--- 8090 T81 BATCH L0346-1.6mm REF (W=760mm)

Fig.2.

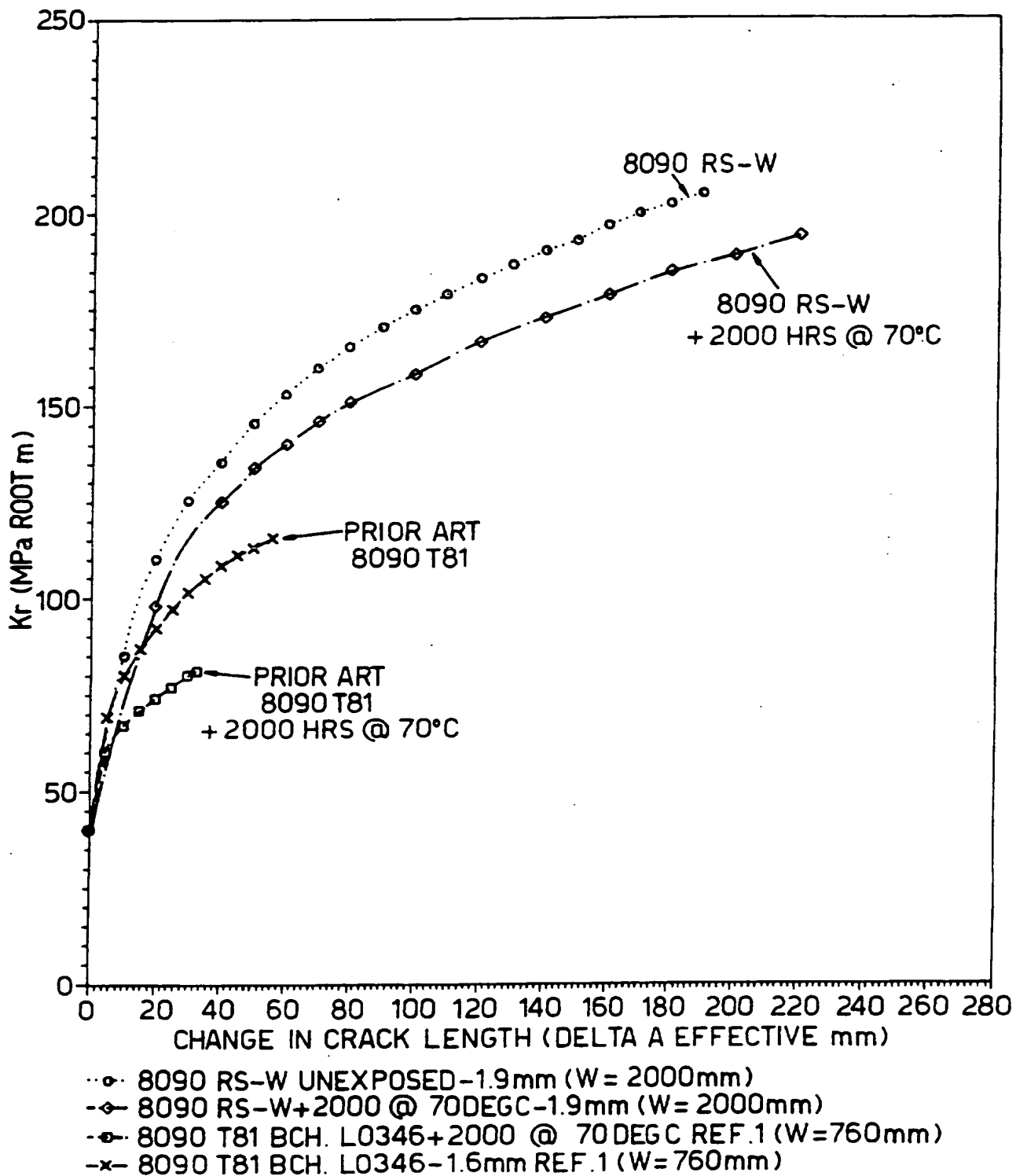
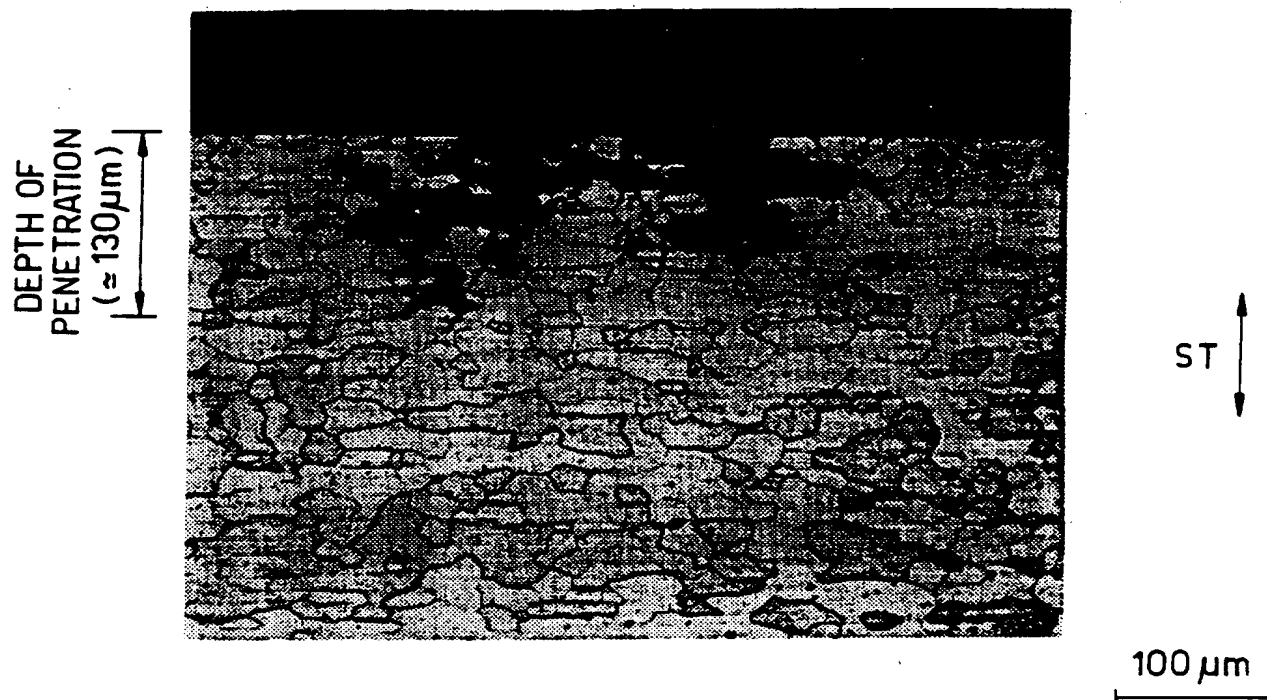
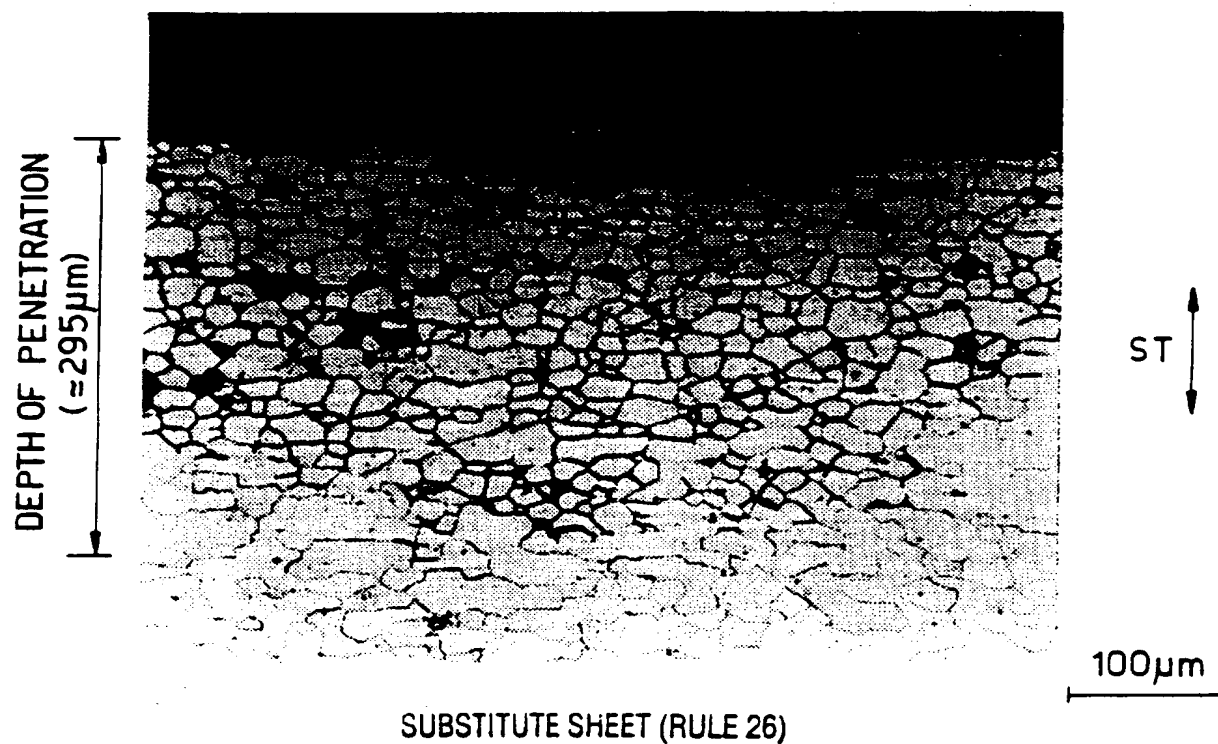


Fig.3.

Fig.4.
PRIOR ART

INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 95/02878

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C22F1/04 C22F1/057

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C22F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	M. PETERS 'ALUMINIUM-LITHIUM PROCEEDINGS 6TH INT. CONF.' 1991, DGM, OBERURSEL, DE XP 000565929 PAGES 137-142 M. FURUKAWA ET AL "SPLIT AGING OF CHILL CAST AL-LI-ZR ALLOYS" see page 138, paragraph 2; figure 2 ---	1
X	WO, A, 91 17281 (ALLIED SIGNAL INC) 14 November 1991 see page 3, line 24 - page 4, line 7 ---	1
X	US, A, 4 812 178 (DUBOST BRUNO ET AL) 14 March 1989 see column 1, paragraph 1 - paragraph 2; claim 1 ---	1
A	EP, A, 0 281 076 (ALUMINUM CO OF AMERICA) 7 September 1988 -----	

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents:

- * "A" document defining the general state of the art which is not considered to be of particular relevance
- * "E" earlier document but published on or after the international filing date
- * "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- * "O" document referring to an oral disclosure, use, exhibition or other means
- * "P" document published prior to the international filing date but later than the priority date claimed

- * "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- * "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- * "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
- * "&" document member of the same patent family

Date of the actual completion of the international search

15 March 1996

Date of mailing of the international search report

26. 03. 96

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+ 31-70) 340-3016

Authorized officer

Gregg, N

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PC1/GB 95/02878

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO-A-9117281	14-11-91	AU-B- 7582291	27-11-91
		CA-A- 2079327	03-11-91
		EP-A- 0528811	03-03-93
		US-A- 5178695	12-01-93

US-A-4812178	14-03-89	US-A- 4897125	30-01-90

EP-A-0281076	07-09-88	US-A- 4790884	13-12-88
		CA-A- 1308630	13-10-92
		DE-A- 3871181	25-06-92
		JP-A- 63235454	30-09-88
